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REPORT R-1615

HYDROGEN EMBRITTLEMENT OF
ELECTROPLATED HIGH STRENGTH STEELS

BY

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AND

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Project No. 31-87 (31-87-1)

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ABSTRACT

The use of high strength steels, free of hydrogen embrittlement, gave rise to an investigation into the effect of hydrogen and the methods of preventing or eliminating embrittlement.

Hydrogen, induced by cathodic charging, cyanide and fluoborate cadmium plating, and chromium plating, was shown to produce embrittlement which varied in severity with the strength level and charging technique in SAE 4340 and 4140 steels. These steels were heat treated to the tensile strength level range between 180,000 and 270,000 psi. The susceptibility to embrittlement increased with increasing tensile strength. Fluoborate cadmium plating did not embrittle as much as other charging methods tested.

Minimum baking treatments to relieve embrittlement were determined for each strength level and charging technique. Static fatigue limits were determined for SAE 4140 steel electroplated with cadmium and chromium. The use of cadmium and electroless nickel preplates as barriers to hydrogen diffusion was proven unsatisfactory as a method of preventing hydrogen embrittlement. An H-11 type tool steel, plated with nickel and cadmium and baked at 600° F, produced an unembrittled corrosion protected steel.

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INTRODUCTION

One of the problems encountered in the use of high strength steels is the severe embrittlement caused by electroplating. Considerable research work has been directed along these lines in the past. The effects of hydrogen on the mechanical properties have been shown by several investigators. ^(1, 2) Delayed failure of embrittled steels under conditions of sustained loading has been studied ⁽³⁾ and the mechanism of delayed failure (concentration of hydrogen at points of triaxial stress, crack initiation, incubation, and propagation) has been investigated. ^(4, 5) Much of the work in the field of hydrogen embrittlement has been summarized in the 1959 Campbell Memorial Lecture by A. E. Troiano. ⁽⁶⁾

There are many instances in the field of U. S. Army Ordnance in which the problem of hydrogen embrittlement assumes considerable importance. Propellant actuated devices are an example of where such problems are encountered. In these devices many components are spring loaded for long periods of time prior to use, and during this time they must be protected from corrosion. Electroplating is preferred for this protection. This is an ideal condition for hydrogen embrittlement to cause failure in the functioning of these parts.

It was the purpose of this investigation to aid in the application of high strength steels to propellant-actuated devices.

MATERIALS

The materials used throughout this investigation were SAE 4140 steel, SAE 4340 steel, and an air hardening type hot work die steel (H-11 Class). The compositions of the various steels are given in Table I.

Table I. COMPOSITION (%) OF VARIOUS STEELS

	Composition (%)		
	SAE 4340	SAE 4140	H-11 (nominal)
C	0.39	0.39	0.40
Mn	0.73	0.88	
P	0.02	0.03	
S	0.016	0.021	
Ni	1.86		
Cr	0.74	0.93	5.00
Mo	0.25	0.23	1.30
Si	0.30	0.28	
V			0.50

The SAE 4340 steel was received as 2-3/8 inch O.D. tubing with a 1/4 inch wall thickness, and was machined into half-ring specimens, as shown in Figure 1A. The SAE 4140 steel was received as 1/2 inch square hot forged rods and was machined into notch tensile specimens, as shown in Figure 1B. The H-11 steel was received as 1/2 inch diameter round bars and was also machined into the notch tensile specimens.

All specimens were heat treated according to the schedule listed in Table II.

Table II. HEAT TREATMENT

	SAE 4340 and SAE 4140	H-11
Normalized	1600° F for 4 hr; air cool	None
Austenitized	1550° F for 1/2 hr	1850° F for 1 hr
Quenched	Oil	Air
Tempered		
Time	2 hr	4 hr
Strength level		
270,000 psi (Rc 50)	550° F	1050° F
230,000 psi (Rc 45)	750° F	-
180,000 psi (Rc 40)	950° F	-

Specimens were ground to final dimensions after heat treatment.

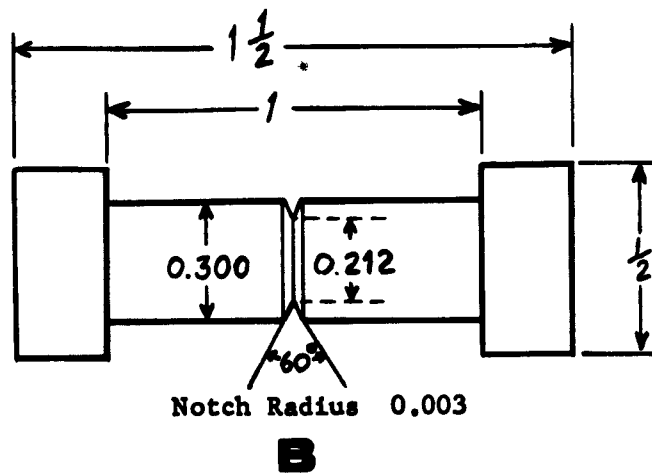
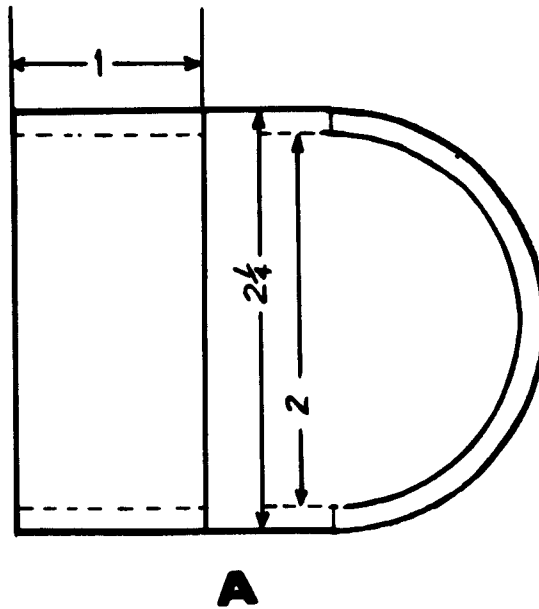


Figure 1. Sketches of
 A - Half-ring Deflection Specimen, SAE 4340 Steel
 B - Notch Tensile Specimen, SAE 4140 Steel

The notch specimens had a finished root radius of 0.003 inch, which resulted in a stress concentration factor of 5.2. ⁽⁷⁾ It was realized that the effects of embrittlement could be better demonstrated using a higher stress concentration factor of approximately 10 (0.001 inch root radius), but due to difficulties encountered in plating the bottom of such a sharp notch, the 0.003 inch root radius was used.

HYDROGEN CHARGING PROCEDURES

Cleaning

Prior to plating or charging, all specimens were cleaned as follows.

1. Lightly sand blasted.
2. Degreased in acetone or trichlorethylene.
3. Alkaline cleaned.

Cathodic Charging

The half-ring specimens were made the cathode in a 4 percent sulfuric acid solution so that hydrogen gas was evolved. A current density of 0.02 amp/sq in. was applied for five minutes.

Cadmium Plating

Cadmium was plated from both cyanide and fluoborate baths. The compositions of the baths are given in Table III.

Table III. CADMIUM PLATING BATHS

<u>Cyanide</u>	
Cadmium oxide (CdO)	26 g/l
Sodium cyanide (NaCN)	130 g/l
<u>Fluoborate</u>	
Cadmium fluoborate $[Cd(BF_4)_2]$	247.5 g/l
Ammonium fluoborate $[NH_4 BF_4]$	90.0 g/l
Harshaw Cadmium Brightener No. 1	12.3 cc/l

A current density of 20 amp/sq ft was used. Specimens were plated for 15 minutes; this resulted in an approximate plate thickness of 0.005 inch. Cadmium anodes were used for both baths.

Chromium Plating

Chromium plating was accomplished using a commercial plating solution* at 125° F and a current density of 2.0 amp/sq in. for a period of 15 minutes. This resulted in a plate thickness of approximately 0.0005 inch. All anodes were made of lead.

Nickel Plating

Several nickel baths were used. The common Watts type bath was used for all electrolytic nickel plating. Electroless nickel plating was produced from two different baths, an alkaline bath with a pH of 9.5 to 10 and an acid bath with a pH of 3 to 5, both at 200° F. The compositions of the baths are listed in Table IV.

Table IV. ELECTROLESS NICKEL BATHS

<u>Alkaline</u>	
Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)	30 g/l
Sodium hypophosphite ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$)	10 g/l
Sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$)	100 g/l
Ammonium chloride (NH_4Cl)	50 g/l
Ammonium hydroxide (NH_4OH)	To adjust pH
<u>Acid</u>	
Nickel sulfate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$)	.25 g/l
Sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$)	3 g/l
Sodium acetate ($\text{NaC}_2\text{H}_3\text{O}_2$)	2.5 g/l
Sodium hypophosphite ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$)	10.0 g/l

* Unichrome C. R. 110 S. R. H. S.

TEST PROCEDURES

Two types of tests were used throughout this investigation: (a) the half-ring compression test, and (b) the notch tensile test. The half-ring compression test was used to determine the degree of embrittlement caused by the various plating methods and the amount of relief which resulted from various baking treatments. From these data, suitable relief treatments were selected and applied to the notch tensile test specimens to determine the relief from embrittlement under conditions of sustained loading.

The strong dependence of hydrogen embrittlement on strain rate and testing temperature has been determined by a number of investigators. (8, 9) It is well known that hydrogen-charged specimens which exhibit embrittlement at moderate strain rates and ambient temperatures can be made to show normal ductilities when tested at high strain rates or low temperatures. Care was taken during the present investigation to hold these variables constant by loading at a constant rate of 0.1 in./min and constant temperature of 70° F.

Half-Ring Compression Tests

The half-ring compression test consisted of compressing a specimen (Figure 1A) across the open diameter and measuring the deflection and load at failure. Tests were performed on specimens which were cathodically charged, fluoborate cadmium plated, cyanide cadmium plated, and chromium plated. Johnson et al⁽¹⁰⁾ have indicated that embrittlement caused by cadmium plating could be prevented by using a thin layer of cadmium as a barrier to the further passage of hydrogen, and plating over this with another layer of cadmium of any thickness. In an attempt to reproduce this result, half-ring specimens were plated according to the following schedule.

1. Plate a thin (0.0001 to 0.0002 in.) layer of cadmium.
2. Bake 1 hour at 350° F to remove hydrogen induced by operation No. 1.
3. Finish cadmium plating to any desired thickness.

The use of nickel, plated from an electroless bath, as a barrier plate was suggested by Beck and Jankowski.⁽¹¹⁾ In an attempt to verify their results, half-ring specimens were plated to a thickness of 0.00016 inch with electroless nickel and then with cadmium, to a total thickness of 0.0005 inch. Both an alkaline and an acid bath were used.

Sustained Load Tests

The sustained load test equipment used was a modification of the Raring and Rinebolt fixture⁽¹²⁾ and is illustrated in Figures 2 and 3. The notch specimen was inserted in a holding fixture which was attached to a proving ring (Figure 2). The proving ring was compressed to a predetermined load. Bolts on either end of the specimen holder were tightened and the compressive load released; this placed the specimen in tension. Strain gages were attached to the rings and calibrated to measure the stress in the specimen.

The loaded rings were placed in a rack (Figure 3) in contact with microswitches which closed when the specimen broke and the ring expanded to its original dimensions. The closing of the switch excited a metering instrument which recorded the time of failure. Strain gage readings were made at intervals during the test to determine if any creep occurred. A maximum creep of 15 microinches per inch in 100 hours was noted. This was equivalent to a drop in stress of 3500 psi. When compared to stresses in the range of 200,000 psi, this was considered small enough to be neglected.

Sustained load tests were used to evaluate the delayed failure properties of the material after the baking treatments were determined by half-ring tests. The properties of the SAE 4140 steel under sustained load, without relief from embrittlement, were determined and expressed in the form of a static fatigue curve. An example of the static fatigue curve, as proposed by Johnson et al, is illustrated in Figure 4.

The sustained load test was also used to evaluate a method of preventing hydrogen embrittlement, as suggested by Beck and Jankowski.⁽¹³⁾ This method consisted of plating a hot work die steel (H-11) with nickel from an electrolytic bath to a thickness of 0.0001 inch, and then plating over this with cadmium to 0.0005 inch total thickness. The steel was baked at 600° F for a sufficient length of time (30 minutes) to relieve embrittlement and allow the cadmium and nickel to diffuse to form an alloy coating. This is a specialized method for H-11 type steels, which can be tempered at these high temperatures.



Figure 2. Notch Tensile Specimen under
Sustained Load in Proving Ring

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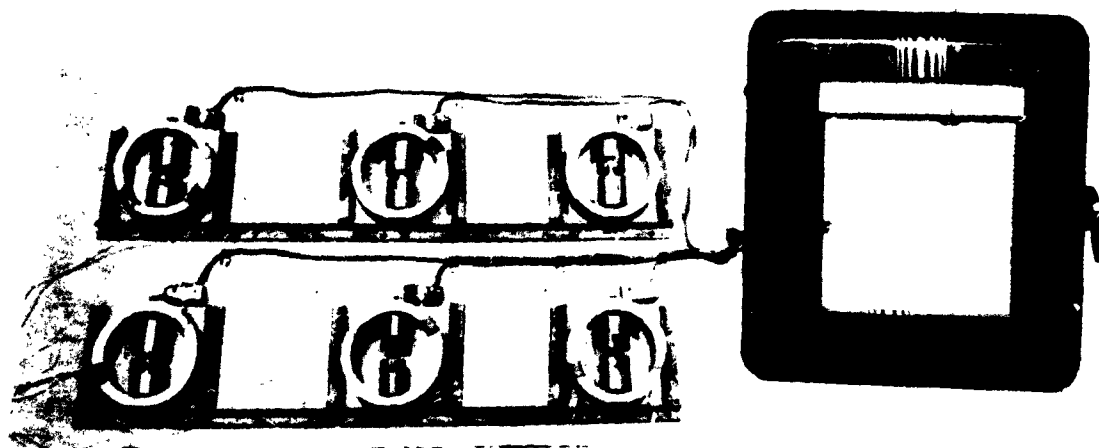


Figure 3. Sustained Load Test Setup for Notch Tensile Specimens

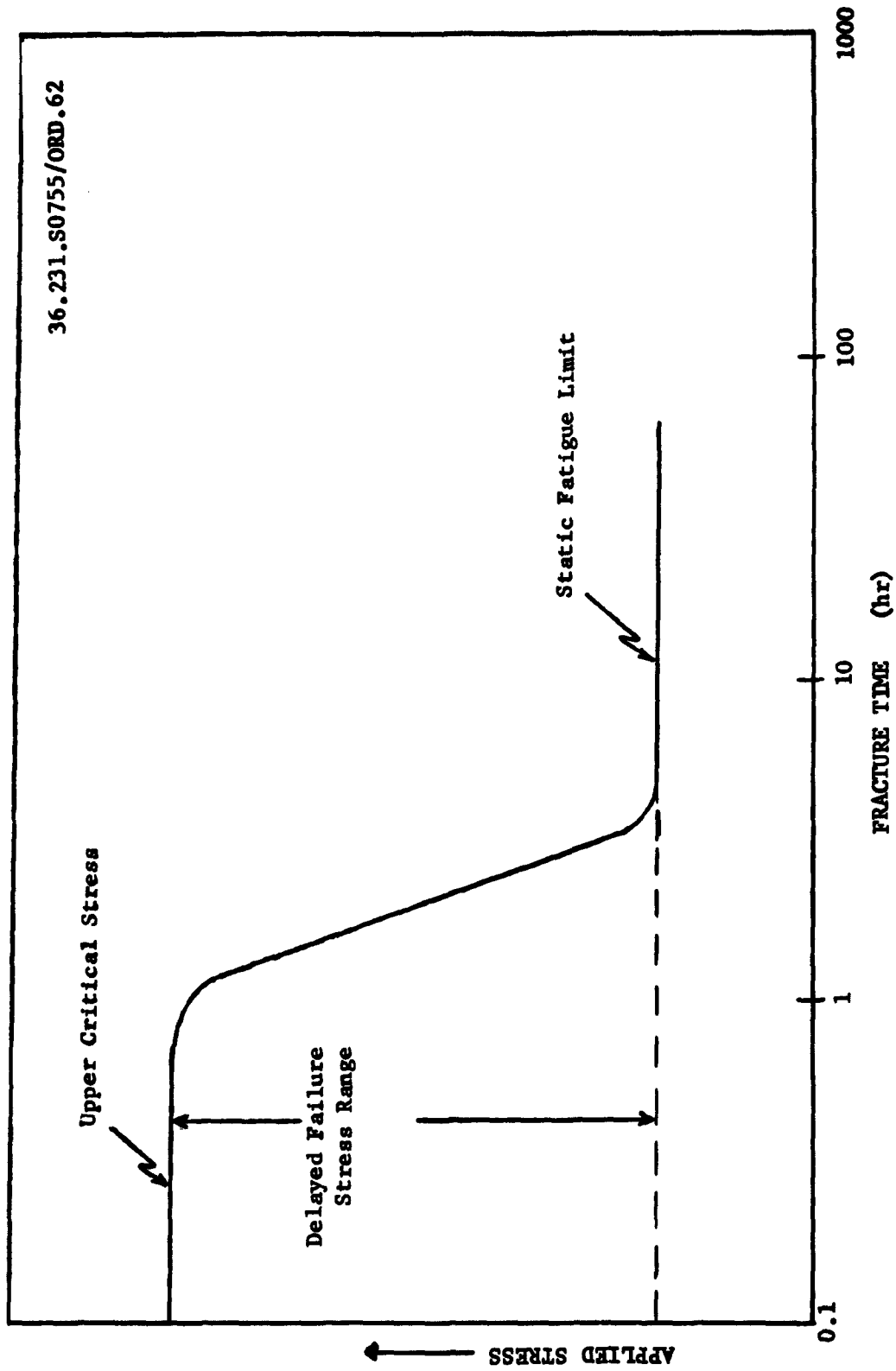


Figure 4. Static Fatigue Curve showing the Characteristics of a Hydrogenated High Strength Steel ("Hydrogen Crack Initiation and Delayed Failure in Steel," by Johnson, Morelet, and Troiano; WADC Technical Report 57-262, May 1957)

RESULTS AND DISCUSSION

Half-Ring Compression Tests

The effect of hydrogen introduced into the specimens by the various methods is shown in Figure 5. These conditions represented the maximum amount of embrittlement for the indicated strength levels since the specimens were tested immediately after charging or plating.

As shown in Figure 5, the condition which exhibited the greatest amount of embrittlement was caused by chromium plating. The fluoborate cadmium plate showed the least amount of embrittlement at the highest strength level tested and was unembrittled at strength levels of 230,000 psi or lower. The chromium and cyanide cadmium plated specimens were both embrittled to a small degree when tested at the 180,000 psi strength level.

Figures 6 through 9 show the results of the aging and baking treatments used to alleviate the embrittled conditions. Appendices A to D inclusive list the numerical data.

As shown in Figure 6, relief from embrittlement caused by cathodic charging of 270,000 psi tensile strength specimens was readily obtained by baking at 250° F or higher for two hours or aging at room temperature for 24 hours. For the 230,000 psi tensile strength specimens, a two-hour age at room temperature was sufficient for relief.

The relief from embrittlement for specimens plated in a fluoborate cadmium bath was achieved by aging at room temperature for two hours or by baking at any of the temperatures shown in Figure 7. The ease of hydrogen removal was expected since the specimens were only embrittled to a small degree initially, as shown in Figure 5.

The embrittlement caused by the cyanide cadmium plating bath was the most difficult to remove. The small degree of embrittlement encountered for the 180,000 psi tensile strength specimens was relieved by baking at 350° F for 4 hours (Figure 8A). Indications were that aging at room temperature for 24 hours would have been sufficient. For the 230,000 psi (Figure 8B) and 270,000 psi (Figure 8C) tensile strength specimens, the minimum practical treatment for relief from embrittlement was 450° F for 4 hours.

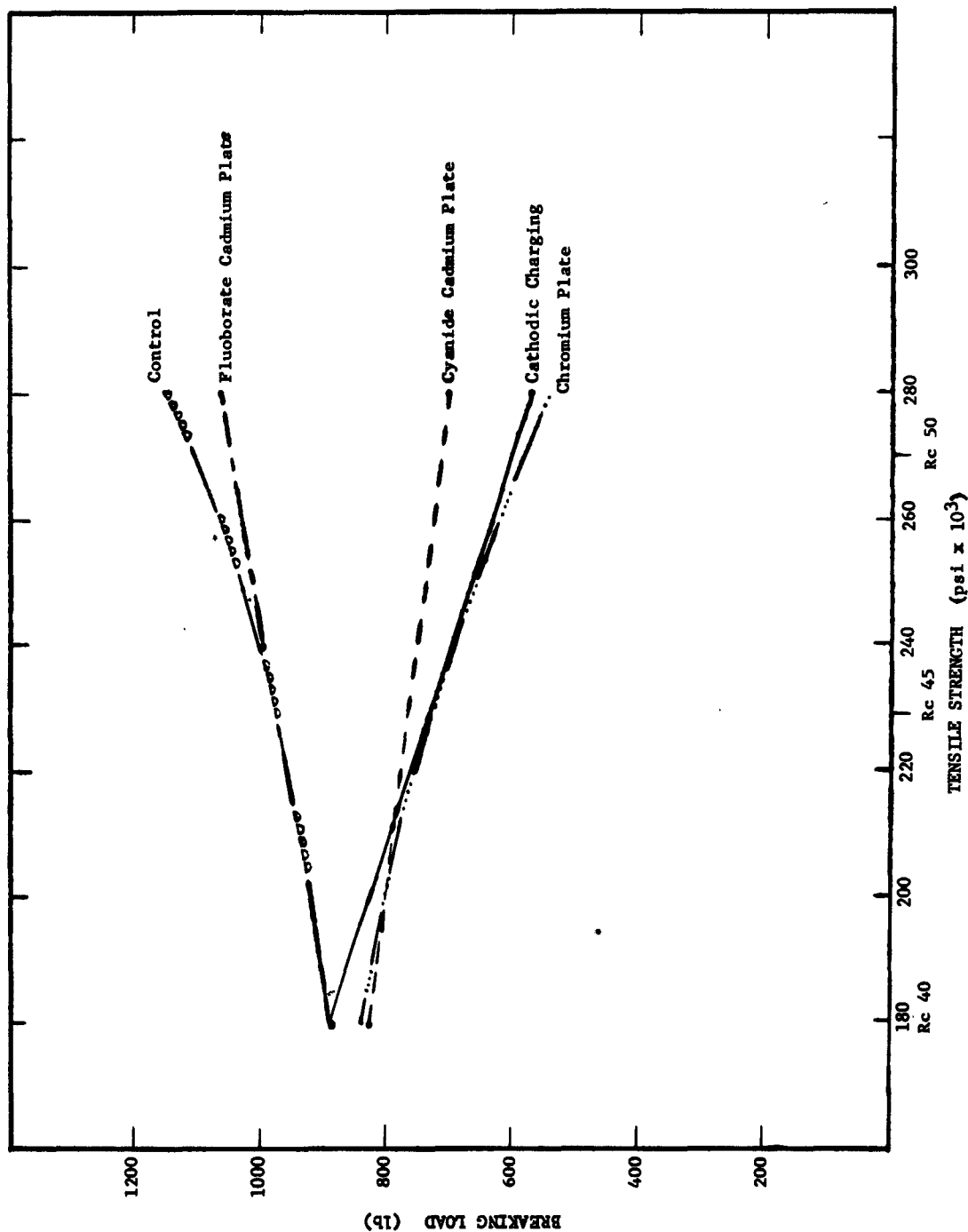


Figure 5. The Effect of Hydrogen induced by various methods on the Breaking Load of Half-ring Specimens (4340 steel) at several Strength Levels

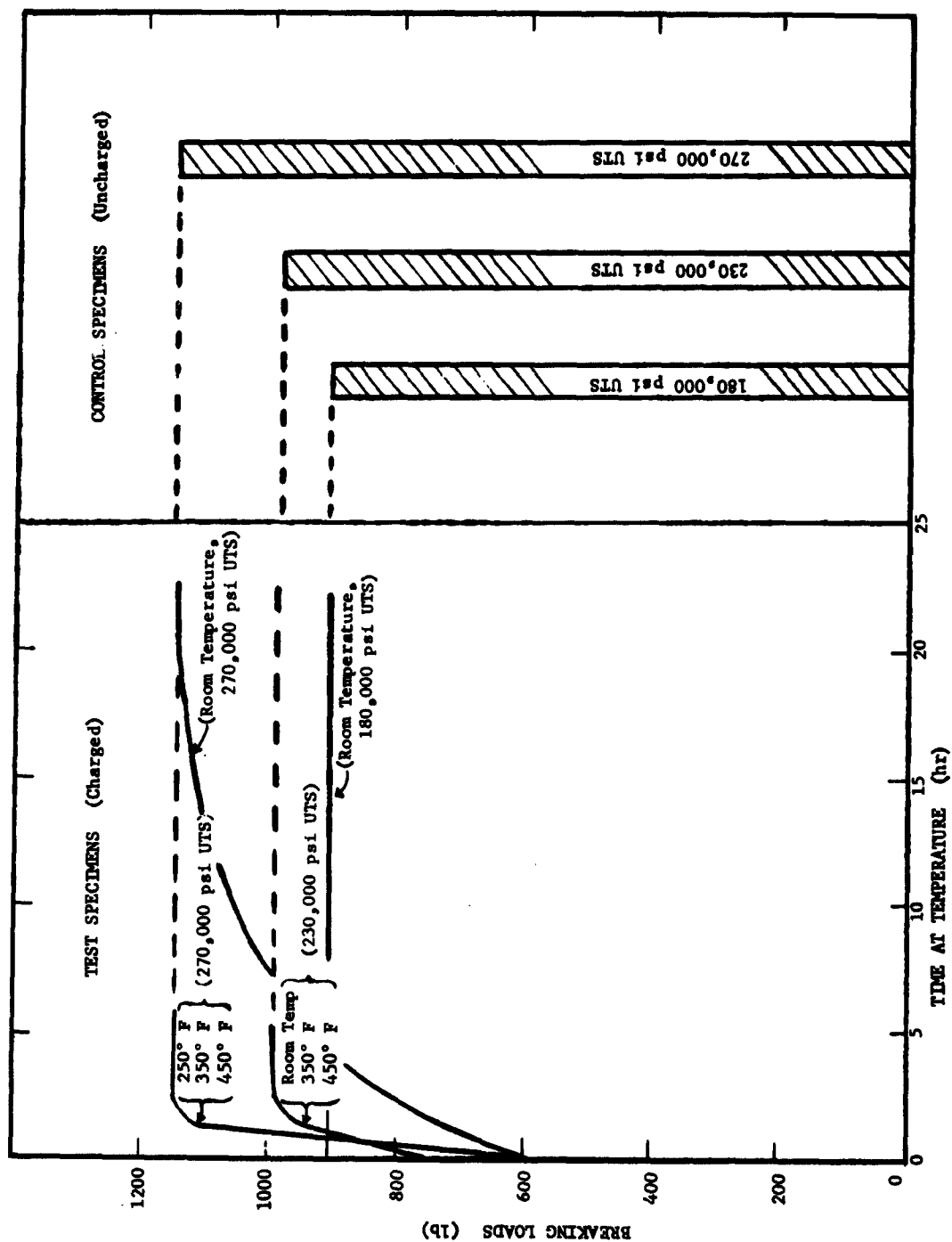


Figure 6. The Effect of Relief Treatments on Breaking Load of Cathodically Charged Half-ring Specimens (4340 steel) at several Tensile Strengths

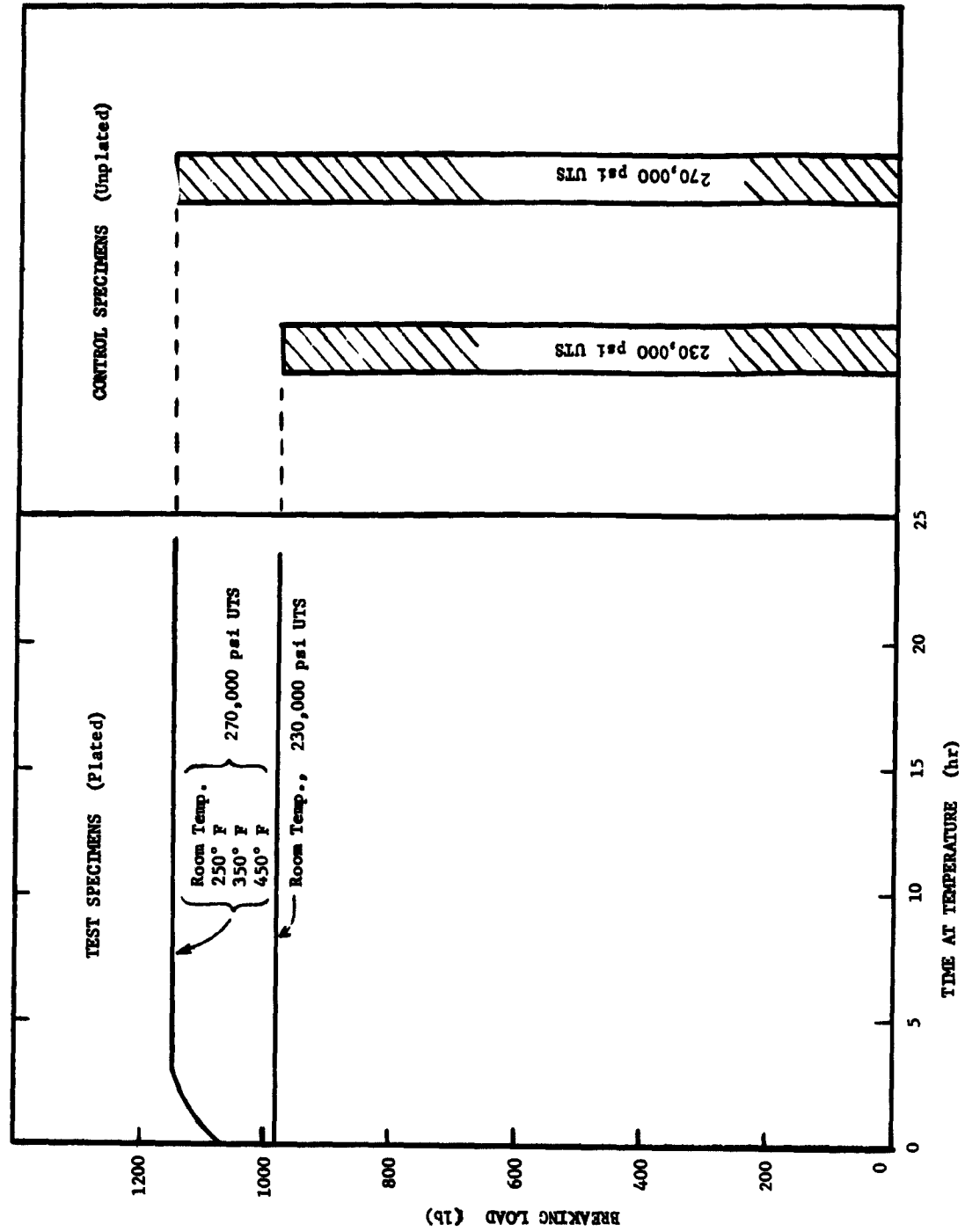


Figure 7. The Effect of Relief Treatments on Breaking Load of Fluoroborate Cadmium Plated Half-ring Specimens (4340 steel) at several Tensile Strengths

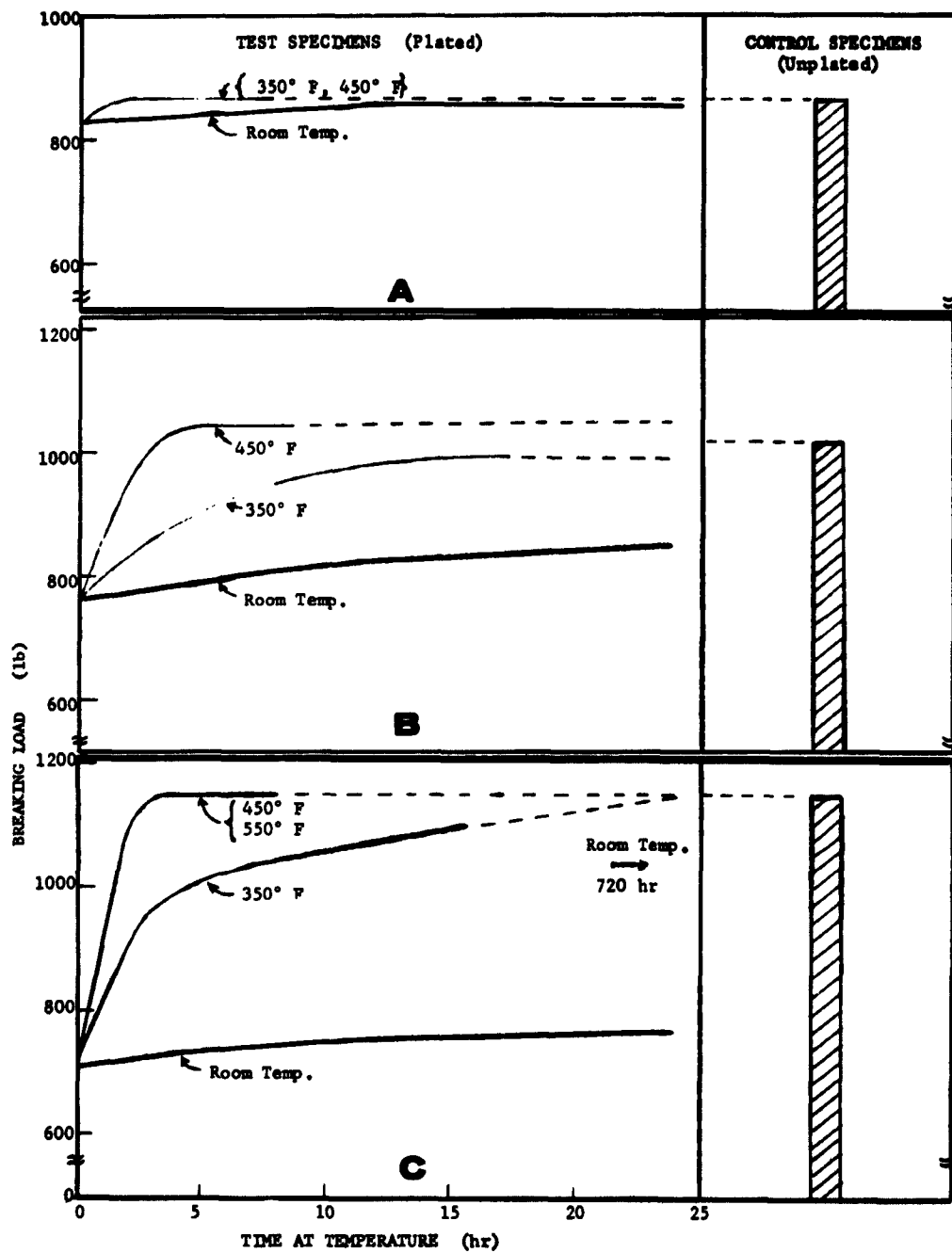


Figure 8. The Effect of Relief Treatments on Breaking Load of Cyanide Cadmium Plated Half-ring Specimens (4340 steel) at
 A - 180,000 psi Ultimate Tensile Strength
 B - 230,000 psi Ultimate Tensile Strength
 C - 270,000 psi Ultimate Tensile Strength

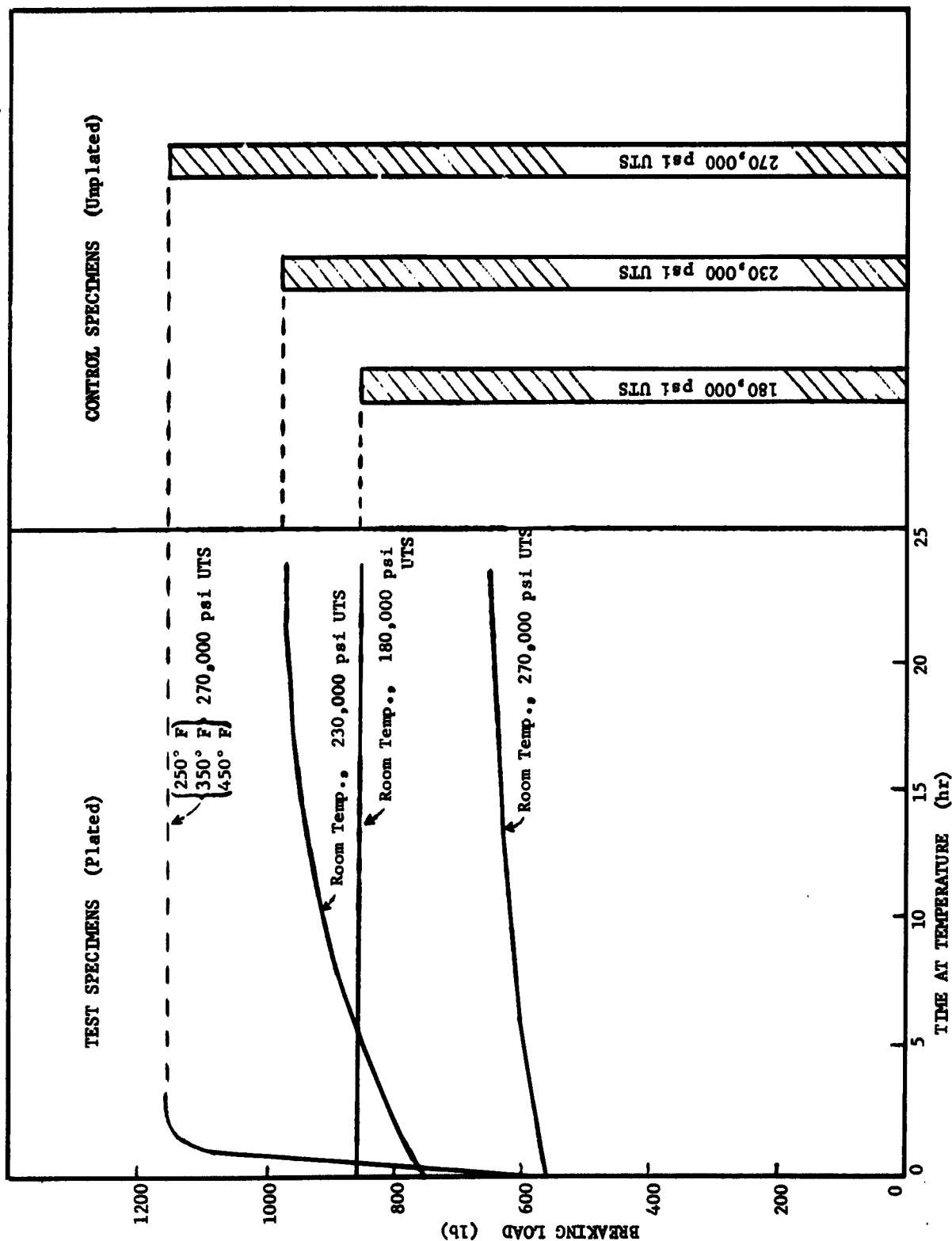


Figure 9. The Effect of Relief Treatments on Breaking Load of Chromium Plated Half-ring Specimens (4340 steel) at several Tensile Strengths

A comparison of the results obtained for cadmium plating from a fluoborate bath and a cyanide bath shows a superiority for the fluoborate bath as far as hydrogen embrittlement is concerned. Unfortunately, the cyanide bath is preferred by electroplaters for its superior "throwing power" and more pleasing appearance. Further development of the fluoborate type cadmium plating should be beneficial to platers concerned with the hydrogen embrittlement problem.

The results obtained with the cyanide cadmium plated specimens show that the treatment to relieve embrittlement of cadmium plated high strength steels listed in Federal Specification QQ-P-416a may not be adequate to remove the effects of hydrogen embrittlement. The specification requires a baking treatment of 3 hours at 375° F for parts heat treated above 230,000 psi tensile strength, while these test results indicate that 4 hours at 450° F is the minimum treatment that will relieve embrittlement.

The results of the chromium plated half-ring tests are shown in Figure 9. Although chromium plating produced the greatest amount of initial embrittlement, it was readily relieved by baking for 2 hours at 250° F.

The relief from hydrogen embrittlement of chromium plated steels listed in Federal Specification QQ-C-320 is a baking treatment for 3 hours at 375° F or 5 hours at 275° F if the 375° F temperature exceeds the tempering temperature. As shown by the test results, either of these treatments would be adequate for relief.

Sustained Load Tests

The treatments for relief of embrittlement of the SAE 4340 steel half-ring specimens were found to be adequate for relief of the SAE 4140 steel sustained load specimens.

The results of the sustained load tests performed on chromium and cadmium plated specimens at the 270,000 psi strength level in the unbaked condition are given by the curves in Figures 10 and 11. The static fatigue curve for the cadmium cyanide plate (Figure 10) shows an upper critical limit of 210,000 psi and a static fatigue limit of 85,000 psi. This results in a stress range of 125,000 psi, in which delayed failure can take place.

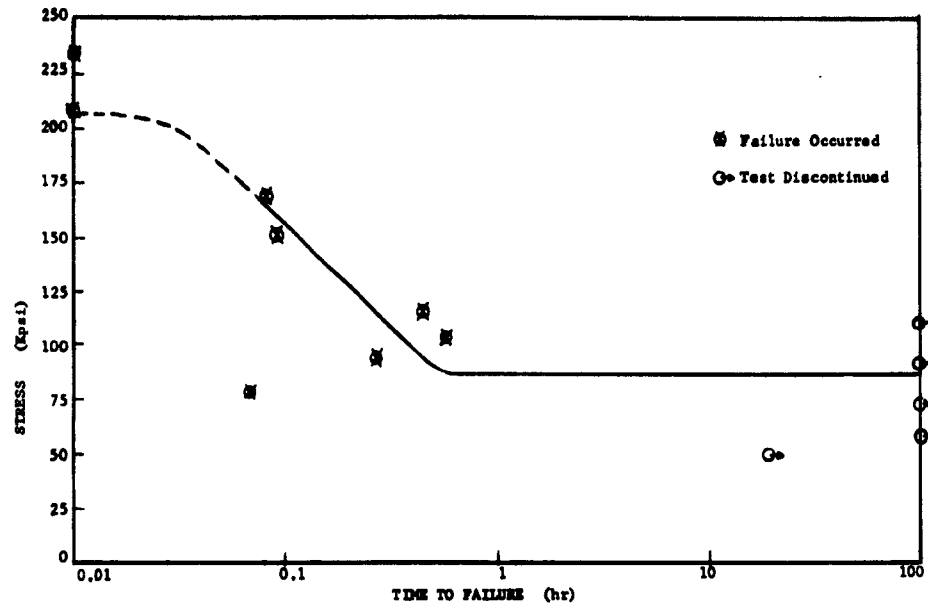


Figure 10. Static Fatigue Properties of Cadmium Cyanide Plated SAE 4140 Steel Notch Tensile Specimens Heat Treated to 270,000 psi Strength Level (Unrelieved)

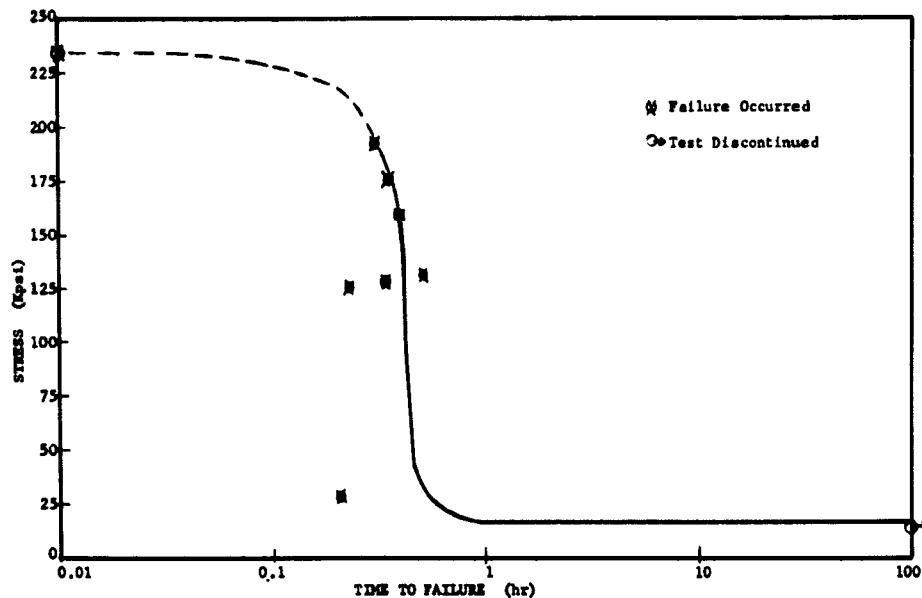


Figure 11. Static Fatigue Properties of Chromium Plated SAE 4140 Steel Notch Tensile Specimens Heat Treated to 270,000 psi Strength Level (Unrelieved)

The static fatigue curve for the chromium plate (Figure 11) shows an upper critical limit of 200,000 psi and a static fatigue limit of 15,000 psi. The delayed failure range is, therefore, 185,000 psi. This is quite large compared to the cadmium plate, but would be expected from the half-ring tests which showed the chromium caused embrittlement to a greater degree than did the cadmium plate.

The sustained load tests were terminated after 100 hours. All test failures which occurred under sustained load usually did so within a limited time period. This does not seem to agree with failures in actual components which occur over longer times under conditions of static load. Some failures which occur in service after longer times under static loads are thought to have the hydrogen supplied by some method other than the manufacturing or plating process.

One theory⁽¹⁴⁾ postulated as to the origin of hydrogen in service failures of cadmium plated parts is that hydrogen may be acquired by the electrochemical action which normally takes place between the cadmium plate and the steel, where the steel is exposed to the atmosphere and cadmium acts as a sacrificial anode. When the electrochemical action takes place, a small amount of hydrogen is evolved. By a process of concentration, this small quantity of hydrogen may be sufficient to embrittle the steel. In recent work, Shank et al⁽¹⁵⁾ have shown that in H-11 steel this type of electrochemical action can produce an average hydrogen content of 2.0 ppm which can be concentrated sufficiently at points of triaxial stress, causing delayed failure.

Prevention of Embrittlement During Plating

Table V shows the results of the barrier plating tests. Although a thin cadmium plate could be baked to render a specimen free of hydrogen embrittlement (Test 1, Table V), the further over-plating of either cadmium or chromium produced embrittlement (Tests 2 and 3). Obviously the cadmium preplate did not act as a barrier to hydrogen diffusion. This result is not in accord with experiments reported in Reference 10.

As shown in Tests 4 and 5, the use of electroless nickel barrier plates, as proposed by the Naval Air Material Center, Philadelphia, Pa., also did not prove effective in preventing hydrogen embrittlement. Later results of their work concurred with these data, and they have withdrawn their claim.

**Table V. RESULTS OF BARRIER PLATING TESTS ON SAE 4340
STEEL HALF-RING DEFLECTION SPECIMENS**

Test No.	Treatment			Results of Test			
	Preplate	Bake (°F)	Overplate	Load		Deflection	
				Pounds	Percent Decrease	Inch	Percent Decrease
1	Cadmium	350	None	1170	0	0.770	0
2	Cadmium	350	Cadmium	1000	13	0.488	34
3	Cadmium	350	Chrome	611	48	0.253	66
4	Alkaline electroless nickel	None	Cadmium	883	23	0.383	49
5	Acid electroless nickel	None	Cadmium	960	17	0.400	46

H-11 steel plated with a layer of nickel, followed by a layer of cadmium, and then baked to diffuse the layers and remove hydrogen, showed no failures when stressed to 300,000 psi for 100 hours. At the high temperatures used for diffusing the nickel-cadmium coating, the hydrogen embrittlement was removed in a relatively short time. For H-11 type steels this is a good method of preventing hydrogen embrittlement.

CONCLUSIONS

1. Results of cathodic charging of SAE 4140 and 4340 steels are:
 - a. Embrittlement occurs above the 180,000 psi strength level.
 - b. The minimum treatment to relieve embrittlement of steels at the 270,000 psi strength level (the maximum tested) is aging at room temperature for 24 hours or baking for 2 hours at 250° F.

2. Results of fluoborate cadmium plating of SAE 4140 and 4340 steels are:

- a. Embrittlement occurs above the 230,000 psi strength level.
- b. The minimum treatment for relief of the 270,000 psi strength level steel is aging 2 hours at room temperature.

3. Results of cyanide cadmium plating of SAE 4140 and 4340 steels are:

- a. Embrittlement occurs at the 180,000 psi strength level and higher.
- b. The minimum treatment for relief of embrittlement at the maximum strength level is 4 hours at 450° F.

4. Results of chromium plating of SAE 4140 and 4340 steels are:

- a. Embrittlement occurs at the 230,000 psi strength level, or higher.
- b. The minimum treatment for relief of embrittlement at the maximum strength level tested is 2 hours at 250° F.

5. The static fatigue limit for SAE 4140 steel without any relief treatment, heat treated to the 270,000 psi strength level, is approximately 85,000 psi for electrodeposited cadmium (cyanide bath) and approximately 15,000 psi for electrodeposited chromium.

6. The use of a thin hydrogen-free barrier plate of cadmium or nickel to inhibit the diffusion of hydrogen from subsequent over-plating operations was unsuccessful.

7. Hot work die steel (H-11) can be plated with cadmium and nickel without being embrittled if heated to a temperature just below the melting point of cadmium. The cadmium and nickel fuse to form a protective alloy coating.

RECOMMENDATIONS

It is recommended that section 3.3.5 of the Federal Specification for Cadmium Plating (QQ-P-416a) be amended to specify a minimum baking treatment of 4 hours at 475° F to insure relief of hydrogen embrittlement.

APPENDIX A

RESULTS OF CATHODICALLY CHARGED HALF-RING DEFLECTION TESTS

Relief Treatment		At 180,000 psi		At 230,000 psi		At 270,000 psi	
Temp	Time	Breaking Load	Deflection	Breaking Load	Deflection	Breaking Load	Deflection
(°F)	(hr)	(lb)	(in.)	(lb)	(in.)	(lb)	(in.)
Control Specimens (uncharged)							
		878	0.876	983	0.773	1154	0.741
Charged Specimens							
Ambient	0	886	0.876	744	0.349	576	0.241
	2			990	0.765	1050	0.606
	4			982	0.755	889	0.418
	8					1034	0.561
	24					1157	0.737
250	2					1153	0.728
	4					1169	0.759
	8					1158	0.740
350	2			983	0.742	1162	0.754
	4			968	0.723	1147	0.724
450	2			981	0.740	1165	0.747
	4			980	0.732	1176	0.747

APPENDIX B

RESULTS OF FLUOBORATE CADMIUM PLATED HALF-RING DEFLECTION TESTS

Relief Treatment		At 230,000 psi		At 270,000 psi	
Temp (°F)	Time (hr)	Breaking Load (lb)	Deflec- tion (in.)	Breaking Load (lb)	Deflec- tion (in.)
Control Specimens (uncharged)					
		983	0.733	1154	0.741
Charged Specimens					
Ambient	0	982	0.735	1065	0.592
	2	996	0.763	1174	0.760
	4			1149	0.720
	24			1179	0.778
250	2	982	0.923	1193	0.777
	4			1179	0.762
350	2			1163	0.737
	4			1166	0.742
450	2			1158	0.737
	4			1173	0.737

APPENDIX C

RESULTS OF CYANIDE CADMIUM PLATED HALF-RING DEFLECTION TESTS

Relief Treatment		At 180,000 psi		At 230,000 psi		At 270,000 psi	
Temp (°F)	Time (hr)	Breaking Load (lb)	Deflec- tion (in.)	Breaking Load (lb)	Deflec- tion (in.)	Breaking Load (lb)	Deflec- tion (in.)
Control Specimens (uncharged)							
		851	0.828	983	0.733	1154	0.741
Charged Specimens							
Ambient	0	828	0.723	769	0.368	708	0.297
	24	860	0.798	845	0.455	770	0.324
	168					702	0.291
	720					1076	0.603
350	2	842	0.768	786	0.345	904	0.458
	4	854	0.803	903	0.720	987	0.488
	8	942	0.821	954	0.665	1041	0.545
	16			979	0.723	1103	0.647
450	2	873	0.845	946	0.649	1104	0.653
	4			958	0.677	1151	0.737
	8			1047	0.755	1154	0.715
550	2			960	0.681	1096	0.653
	4			960	0.706	1154	0.744

APPENDIX D

RESULTS OF CHROMIUM PLATED HALF-RING DEFLECTION TESTS

Relief Treatment		At 180,000 psi		At 230,000 psi		At 270,000 psi	
Temp (°F)	Time (hr)	Breaking Load (lb)	Deflection (in.)	Breaking Load (lb)	Deflection (in.)	Breaking Load (lb)	Deflection (in.)
Control Specimens (uncharged)							
		851	0.828	968	0.745	1154	0.741
Charged Specimens							
Ambient	0	837	0.755	742	0.341	561	0.239
	24			965	0.713	653	0.281
250	2			989	0.746	1147	0.729
350	2					1161	0.765
450	2					1155	0.735

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